High-Temperature Pressure Vessel Reactor

User's Manual

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APPLIED PHOTONICS

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1 Introduction

The four High-Temperature Pressure Vessel Reactor units to which this manual refers were designed and manufactured by Applied Photonics Ltd for Nexia Solutions Ltd under contract number 4500384268 (2 Dec 2004 to 31 Jan 2005) and contract number 4500413679 (21 March to 15 April 2005). The units were designed to a general specification provided by Nexia Solutions Ltd and are suitable for use on an open bench, inside a fumehood or enclosed within a glovebox. Each unit has two independent pressure relief devices to prevent over-pressurisation of the vessel (a relief valve and a precision burst disc) and two independent methods of monitoring the internal pressure (an electronic pressure transducer and an analogue pressure gauge). For glovebox use, the units have been designed so that only partial disassembly is required for posting through a 10" diameter glovebox port. In consideration of the special requirements of a glovebox environment, a number of additional safety features have been included to minimise the risk of i) over-pressurising the glovebox in the event of one or both of the safety relief valves venting gas from the pressure vessel, and ii) thermal damage to heat-sensitive glovebox components, in particular the polymer gloves. These additional safety features are described in the following sections of this manual.

This User's Manual should be read in conjunction with the Technical File provided to Nexia Solutions Ltd with the High-Temperature Pressure Vessel units. The Technical File provides complete design information for the units including detailed engineering drawings, assembly instructions for certain sub-assemblies such as the heating mantle, electrical drawings, general arrangement drawings / images, and certificates of conformity for certain components and devices. A comprehensive parts list is also included with the Technical File.

2 Safety

2.1 Electrical

Certain parts of the High-Temperature Pressure Vessel Reactor (ie. the controller and the heating mantle) contain electrical components operating at potentially lethal voltage and current levels. Always unplug the instrument from the mains supply before carrying out any servicing of the instrument. Servicing of the instrument should be carried out only by suitably qualified personnel. The instrument is designed as a Class 1 electrical product and so must be connected to a safety earth ground via the mains supply lead. For added safety, it is highly recommended that the controller is connected to the 230 VAC mains supply using the lead provided with the controller and which is fitted with a 30 mA Residual Current Device (RCD). It is recommended also that the controller is only connected to the mains supply when the controller is properly connected to the heating mantle using i) the supplied flexible power cable terminated with Lemo plug and socket, and ii) the thermocouple cable – proper connection is described later in this manual.

Note on Portable Appliance Testing
The cartridge heaters used in the heating mantle have a relatively high leakage current to earth, the exact figure varying from heater to heater. In view of this, an insulation impedance test could yield a value of as low as 0.3 MΩ. This value is acceptable and has been taken into account in the electrical design of the units.

2.2 High pressures and temperatures

All pressures mentioned in this User's manual are gauge pressures. The pressure vessel and associated pipework are designed to operate, under normal operating conditions, at pressures of up to **30 Bar** at any temperature in the range ~20°C (room temperature) to the maximum operating temperature of 600°C. In order to allow for a significant safety margin, the maximum design pressure (ie. Safe Working Pressure) of the vessel is 50 Bar @ 600ºC. Two independent pressure limiting devices are included in the design of

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the pressure vessel; a burst disc which is designed to open if the pressure exceeds 37 Bar and a pressure relief valve which may be set by the user to any pressure in the approximate range 20 Bar to 50 Bar.

Warning

If the system is pressurised to greater than ~30 Bar, the burst disc will be weakened which could result in the burst disc activating (ie. bursting) at pressures less than the rated burst pressure of 37 Bar. Activation of the burst disc will require a replacement to be fitted. It is essential that only the same type and rating of burst disc is fitted - contact the manufacturer if a replacement burst disc is required.

The outlet of both the burst disc and pressure relief valve are fitted with a sintered metal filter (0.5 micron pore size). The purpose of the filters is twofold; i) to prevent particulates escaping from the pressure vessel, and ii) to limit the flow-rate of gas escaping from the pressure vessel (ie. to reduce the risk of over-pressurising the glovebox in which the unit will ultimately be located) when either safety device operates.

The heating mantle is designed to heat the pressure vessel to temperatures of up to 600°C. It is therefore clear that the pressure vessel and other components in close proximity could be at elevated temperatures and hence pose a risk to the user of burns to the skin should contact be made with these components. A guard is supplied with the unit which is designed to prevent accidental contact with the hot components. It is important that this guard is fitted to the unit whenever the pressure vessel is to be operated at elevated temperatures. The pressure vessel will take a considerable period of time to cool from elevated temperatures. Before starting to dismantle the pressure vessel body, it is vital that the user checks that the vessel is not at pressure and/or elevated temperature.

Warning

To prevent damage to the units and to minimise the risk of personal injury, it is the responsibility of the user to ensure that the following ratings are not exceeded:

Pressure Vessel Maximum Operating Pressure = 30 Bar (gauge)

Pressure Vessel Maximum Operating Temperature = 600°C

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3 General description

3.1 High-temperature pressure vessel and support stand assembly

The support stand provides a platform on which the pressure vessel head, associated pipework and pressure components (safety relief valves, pressure gauges, inlet/outlet ports, etc) are located. The mechanical attachment to the pressure vessel head is designed for adequate strength whilst maintaining a relatively low thermal conduction path between the vessel head and the support stand assembly. The pressure vessel body is fitted to the vessel head using high-tensile (black steel) M8 x 40 hex head cap screws (8-off). Two designs of pressure vessel body are included with each unit; the first (Pt. No. 1.01) is designed to accommodate a silver-plated C-ring metal seal (Pt. No. 2.01) and the second (Pt. No. 1.02) is designed to accommodate either a PTFE seal (Pt. No. 1.04) or a nickel-plated C-ring seal (Pt. No. 2.81). The PTFE seal is suitable for vessel temperatures up to approximately 225°C whereas either of the metal C-ring seals are suitable for temperatures up to the maximum rating for the pressure vessel (ie. 600°C). The PTFE seal is re-useable to a limited extent whereas the metal C-ring seals are designed for single-use only.

The support stand includes a lifting mechanism which is used to manually lift the heating mantle into position and lock it in place. Further information on the correct procedure for securing the heating mantle to the pressure vessel is given in section 4.2.

General views of the high-temperature pressure vessel and support stand assembly are given in figures 1 to 5.

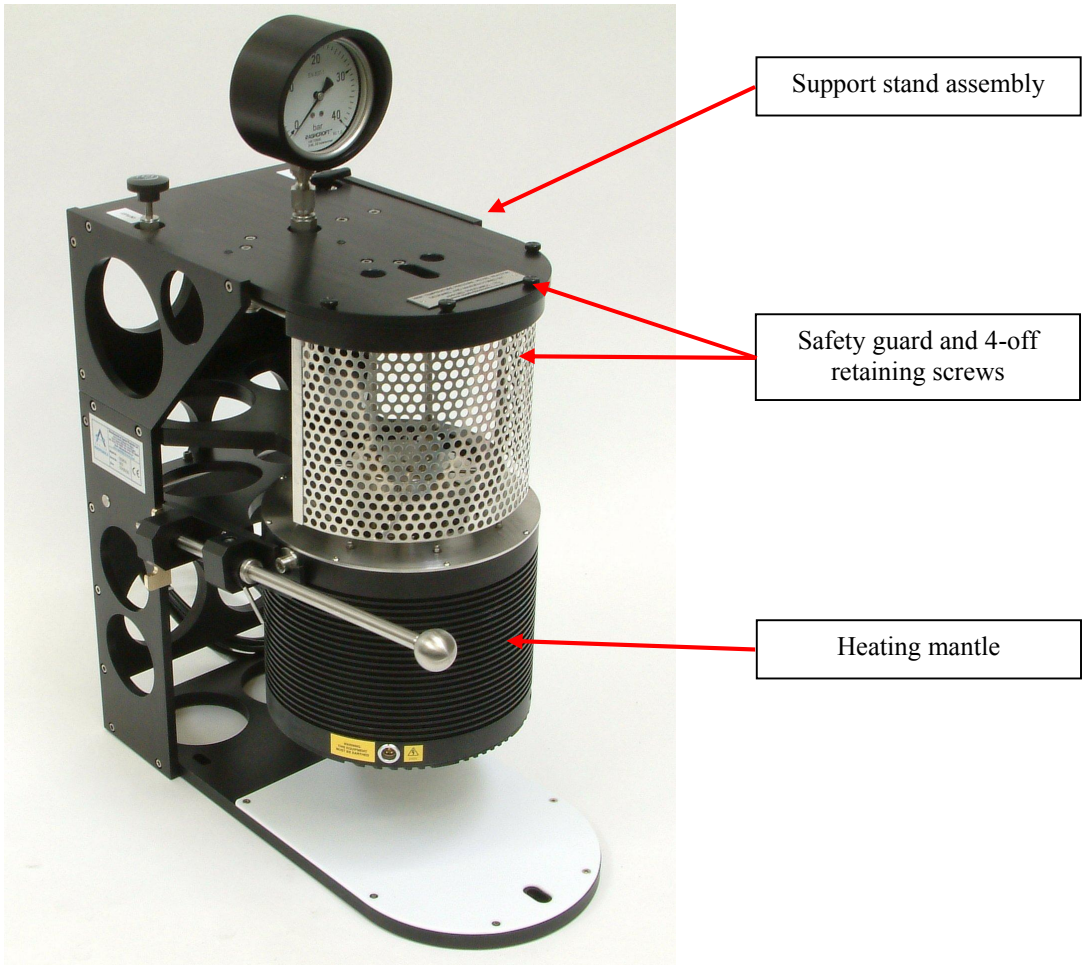

Figure 1. General view of the high-temperature pressure vessel unit

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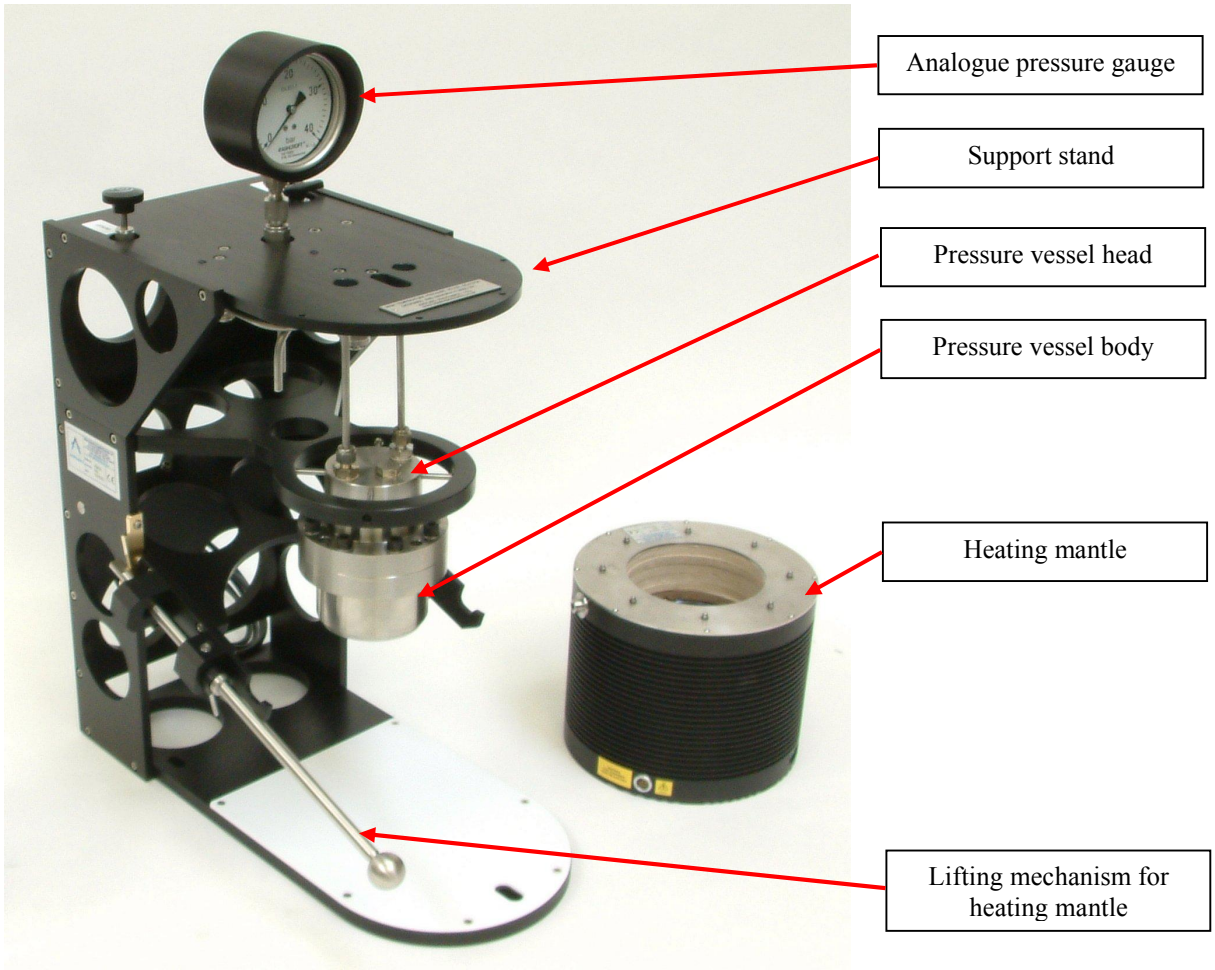

Figure 2. General view of the high-temperature pressure vessel unit with heating mantle removed.

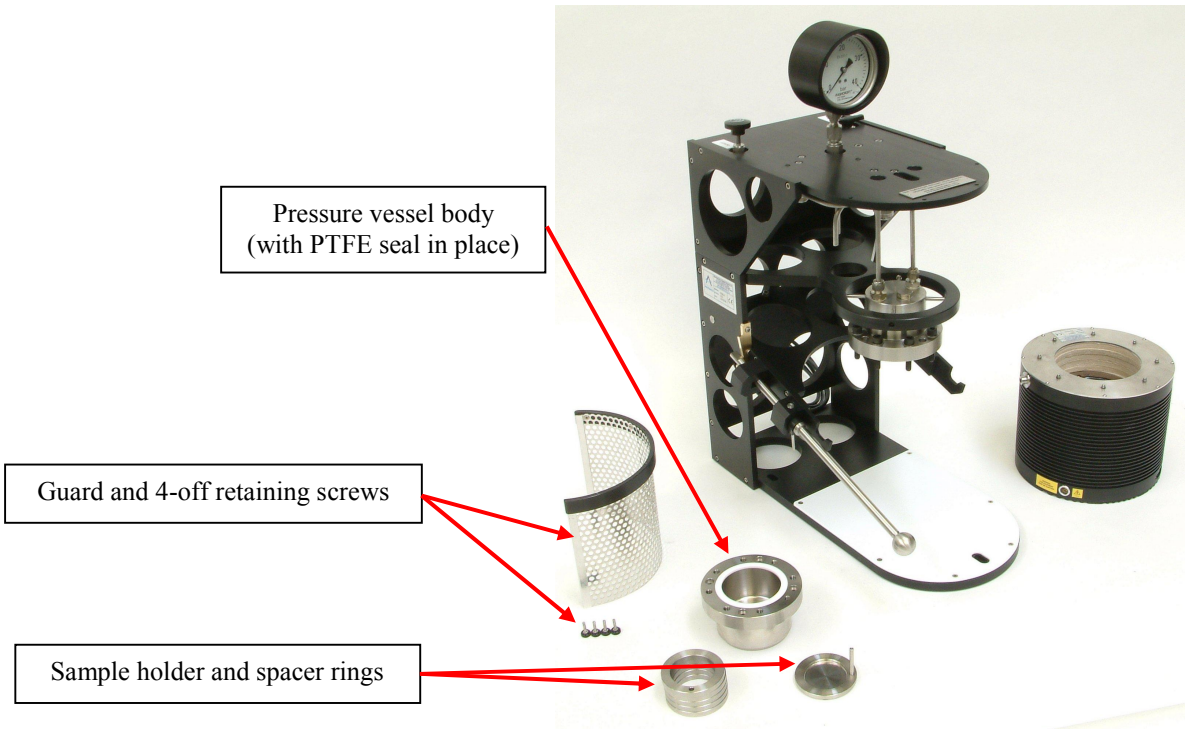

Figure 3. General view of the high-temperature pressure vessel unit with vessel body removed.

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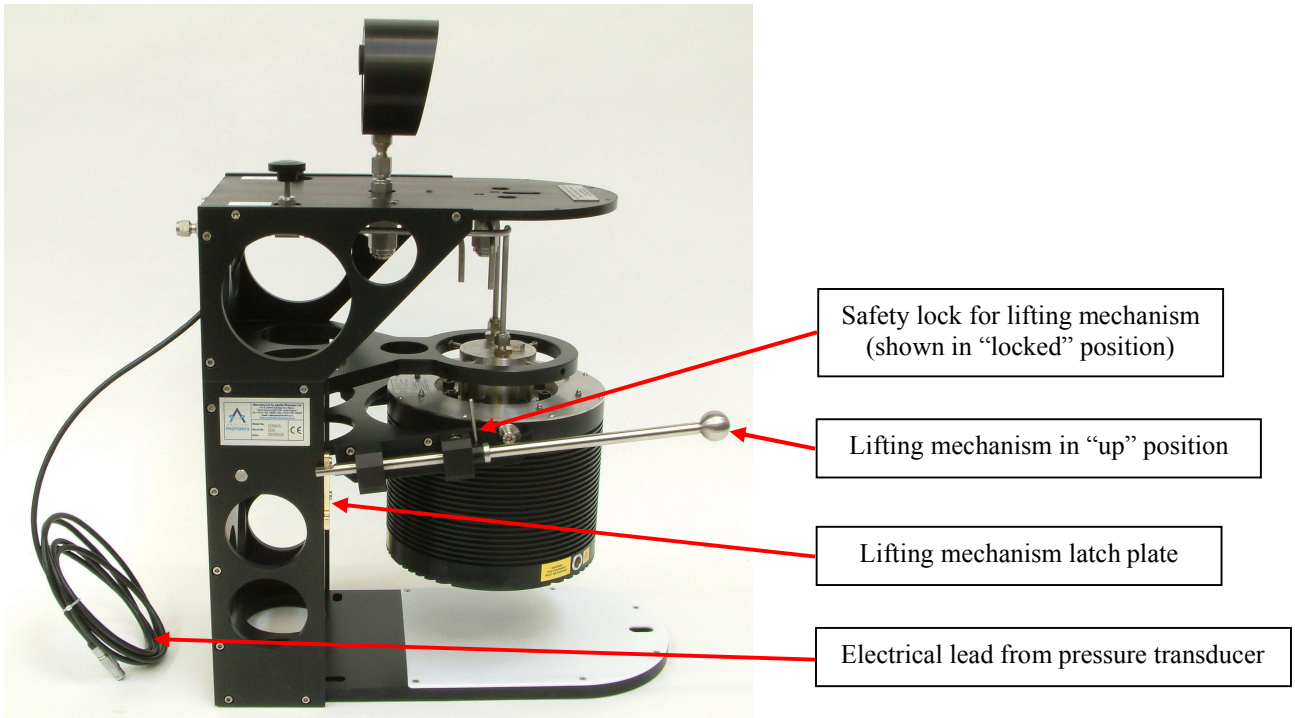
Safety lock for lifting mechanism (shown in “locked” position)
Lifting mechanism in “up” position
Lifting mechanism latch plate
Electrical lead from pressure transducer

Figure 4. Left side view of unit with heating mantle fitted

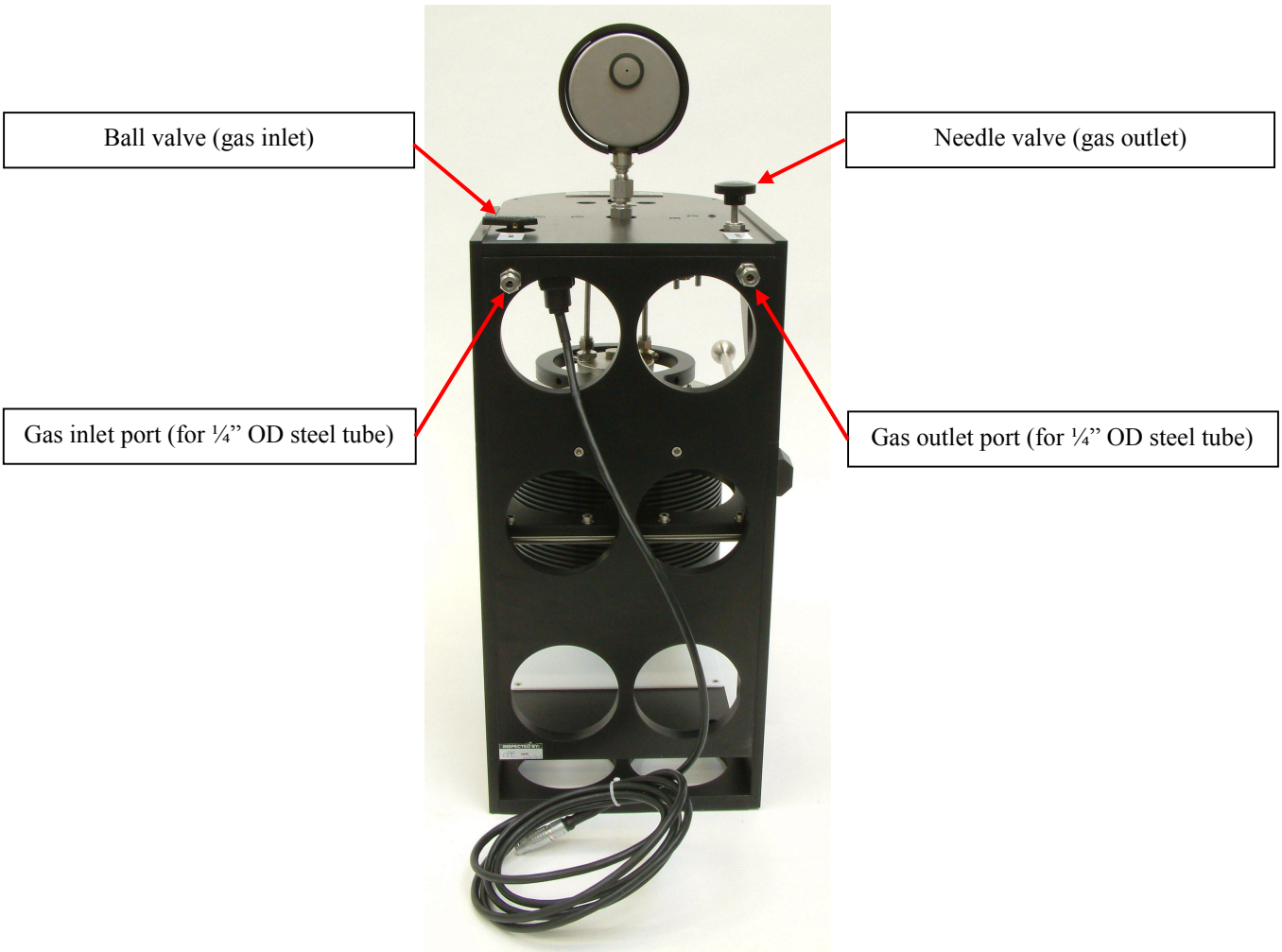

Figure 5. Rear view of unit

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3.2 Heating mantle

The design and assembly of the heating mantle is described in detail in the Technical File supplied separately to this User's Manual. In brief, the heating mantle consists of a stainless steel heater body located in a thermally insulated mantle within an outer aluminium can. The heater body contains six cartridge heaters each rated at 150 Watts, 230 VAC, giving a total power of 900 Watts. One of the cartridge heaters contains an embedded Type K thermocouple, connection to which is made via the thermocouple chassis socket located as shown in figure 6. Electrical power to the cartridge heaters is made via the power socket located as shown in figure 6. The outer aluminium can is finned and anodised black to help to dissipate heat and hence maintain a relatively low temperature. When operating the heating mantle at temperatures of greater than approximately 250°C, it is advisable to provide forced-air convection to help dissipate heat build-up in the outer aluminium can. Tests have shown that with only a modest air-flow, the temperature of the outer aluminium can remains less than 40°C even when the heating mantle is operated at its maximum rated temperature of 600°C for prolonged periods. To prevent over-heating of the heating mantle, two thermal fuses wired have been included in the design, both of which are in thermal contact with the outer aluminium can. Activation of either or both thermal fuses will interrupt electrical power to the cartridge heaters. One thermal fuse is of the "automatic reset" type and is designed to cut electrical power to the heaters when the temperature of the outer aluminium can reaches 70°C and reconnect power when the temperature falls to 55°C. The second thermal fuse, which acts as a back-up in the event of failure of the first thermal fuse, is of the "manual reset" type and is designed to permanently cut electrical power to the heaters when the temperature of the outer aluminium can reaches 100°C. Should this fuse be activated, it will be necessary to return the heating mantle to the manufacturer for inspection and servicing.

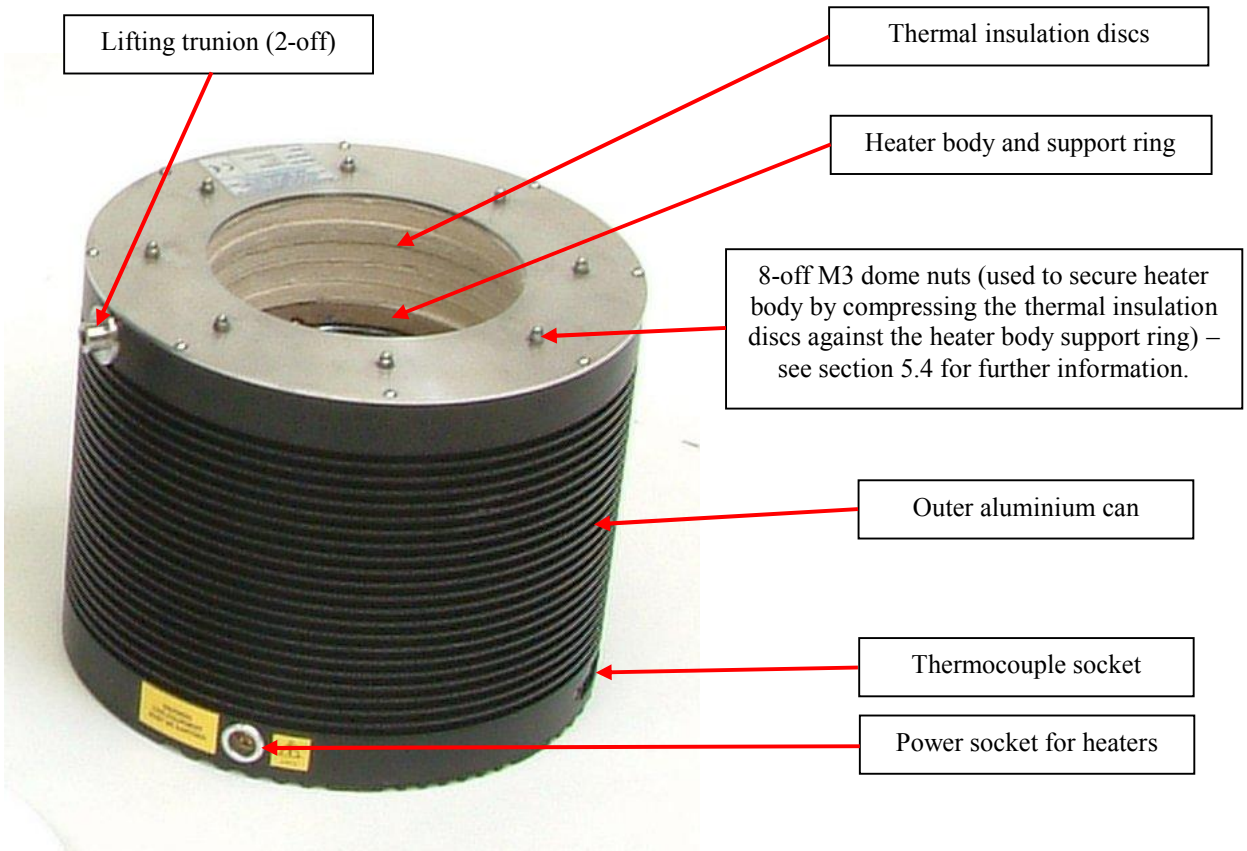
Lifting trunion (2-off)
Thermal insulation discs
Heater body and support ring
8-off M3 dome nuts (used to secure heater body by compressing the thermal insulation discs against the heater body support ring) – see section 5.4 for further information.
Outer aluminium can
Thermocouple socket
Power socket for heaters

Figure 6. Image of heating mantle

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3.3 Pressure Vessel Controller (PVC)

The Pressure Vessel Controller (PVC) monitors and controls the temperature of the heaters in the heating mantle and, if required, the temperature of the gas within the pressure vessel by means of an additional (optional) thermocouple probe. The optional probe (Pt. No. 2.17) is inserted into the pressure vessel using the spare ¼" BSPP port in the head of the vessel and the high-temperature sealing gland (Pt. No. 2.16). The PVC also monitors the pressure of the gas within the vessel via the electronic pressure transducer.

The PVC may be configured in either of two operating modes – see section 4.3 for further details on this.

The PVC contains two fuses. The main fuse (5 Amp) is in the IEC switched mains input socket located on left of the rear panel (see figure 8). Another fuse (1 Amp, Quick Blow) is used to protect the electronic devices within the PVC and is located inside the PVC enclosure in a fuseholder on the DIN rail (see figure 13, page12). Replacement fuses must be of the same type and rating.

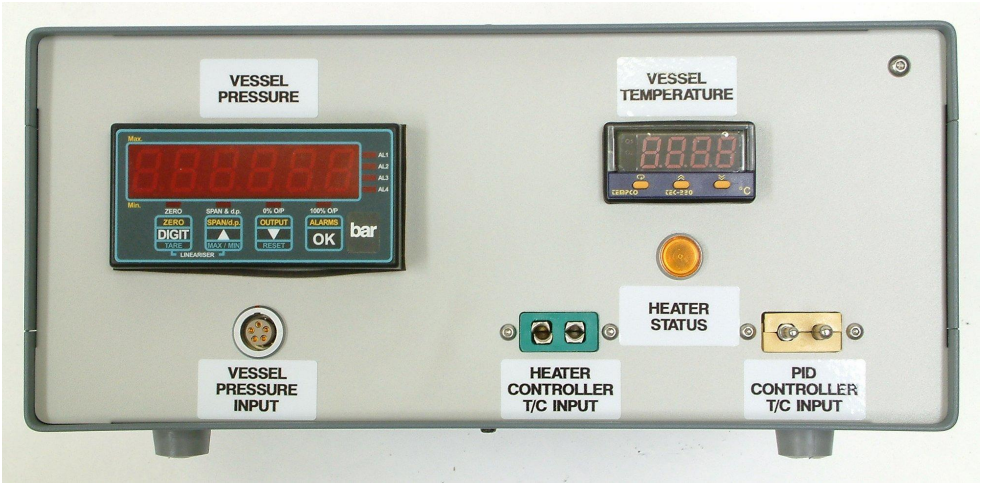

Figure 7. Image of front view of PVC

Figure 8. Image of rear view of PVC

Warning

The Pressure Vessel Controller MUST be connected to a protective earth via the mains power lead. It is highly recommended also that the RCCD protected power lead supplied with the unit is used.

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4 Operating procedures

4.1 Fitting the pressure vessel body

Two types of pressure vessel body are supplied with each unit; vessel body (Pt. No. 1.01) which utilizes a silver-plated C-ring seal (Pt. No. 2.01) and vessel body (Pt. No. 1.02) which utilizes either a PTFE seal (Pt. No. 1.04) or a nickel-plated C-ring seal (Pt. No. 2.81). Before fitting the vessel body to the vessel head, it is important to ensure that the mating surfaces are scrupulously clean and the correct sealing gasket is fitted. The sample holder and, if required, volume-reducing spacers, should be fitted inside the vessel body (see figure 9). Lift by hand the vessel body and mate it up to the lower flange face of the vessel head ensuring that the 4-off counter-bored M5 holes in the body flange align with the 4-off M5 clearance holes in the vessel head flange. Secure the vessel body to the head using 8-off M8 x 40 high-tensile (black steel) hex head cap screws and M5 s/s plain washers (see figure 10). Tighten the M8 screws progressively and evenly until the flanges faces meet (this will be obvious by a sudden increase in the torque required to tighten the screws). Do not tighten further. While tightening these screws, it is recommended that the special C-spanner (Pt. No. 1.39) is used to prevent excess torque being transferred to the 4-off vessel head securing pins.

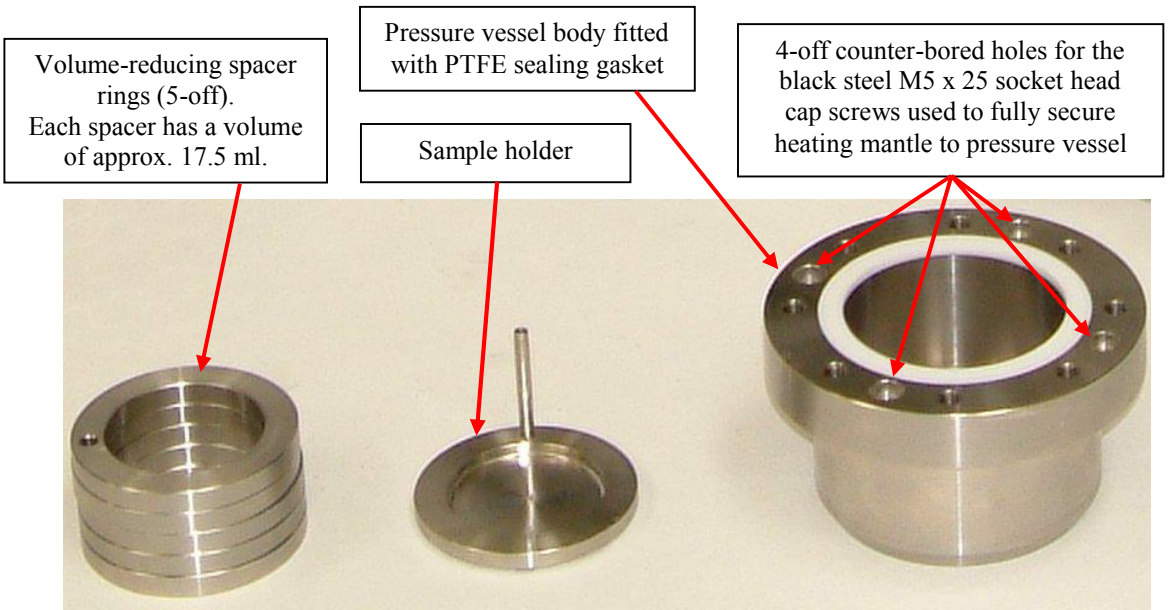
Volume-reducing spacer rings (5-off). Each spacer has a volume of approx. 17.5 ml.
Pressure vessel body fitted with PTFE sealing gasket
Sample holder
4-off counter-bored holes for the black steel M5 x 25 socket head cap screws used to fully secure heating mantle to pressure vessel

Figure 9. Image of pressure vessel body, sample holder and volume-reducing spacer rings

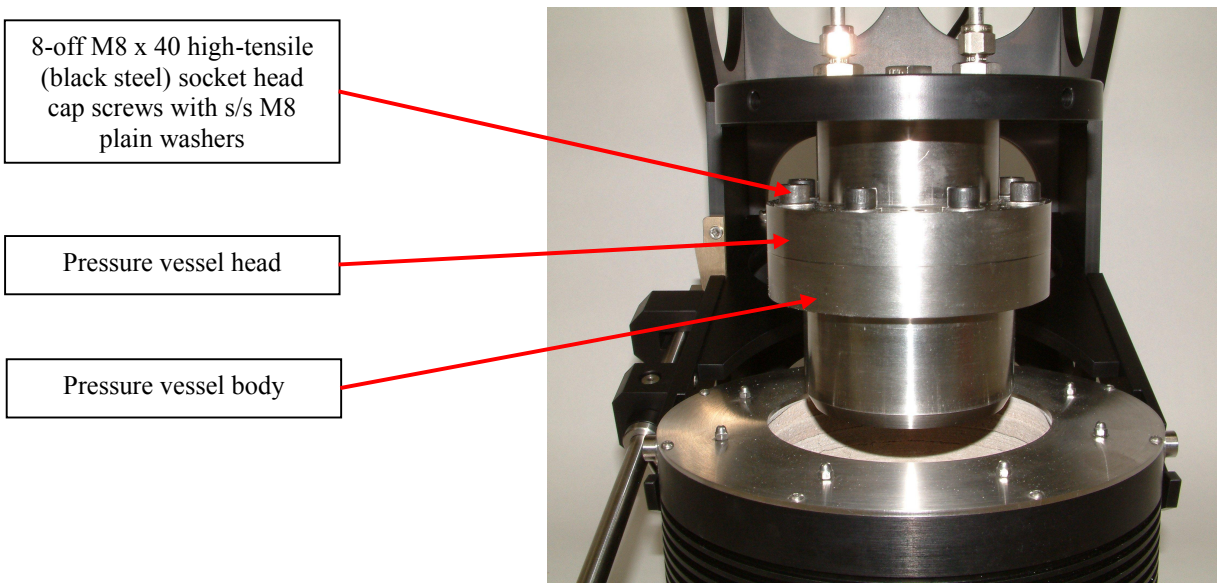
8-off M8 x 40 high-tensile (black steel) socket head cap screws with s/s M8 plain washers
Pressure vessel head
Pressure vessel body

Figure 10. Image of pressure vessel body fitted to vessel head

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4.2 Fitting the heating mantle to the pressure vessel

Lower the lifting mechanism to the "down" position. Place the heating mantle on the PTFE base plate cover with the power socket and thermocouple socket facing forwards as indicated in figure 11. Slide the heating mantle back so that the two lifting trunions locate in the yoke of the lifting mechanism. Using the handle of the lifting mechanism, gently raise the heating mantle while guiding it by hand so that the heater body slides over the pressure vessel body. When the lifting mechanism is in the fully up position, the lifting handle will spring in to the upper recess of the latch plate to maintain the heating mantle in the up position. A safety lock located on the lifting handle (see figure 12) may be used to prevent accidental dislodging of the lifting mechanism. Using the 4-off black steel M5 x 25 hex head cap screws provided with the unit, secure the heating mantle to the pressure vessel by inserting the screws from above into the clearance holes in the flange of the pressure vessel head. These screws only need to be tightened sufficiently to hold the heating mantle firmly in place - do not overtighten these screws as there is a risk that they may seize in place when the unit is operated at elevated temperatures.

Warning

Do not use stainless steel screws for securing the heating mantle to the pressure vessel as they are highly likely to seize in place making removal of the heating mantle impossible.

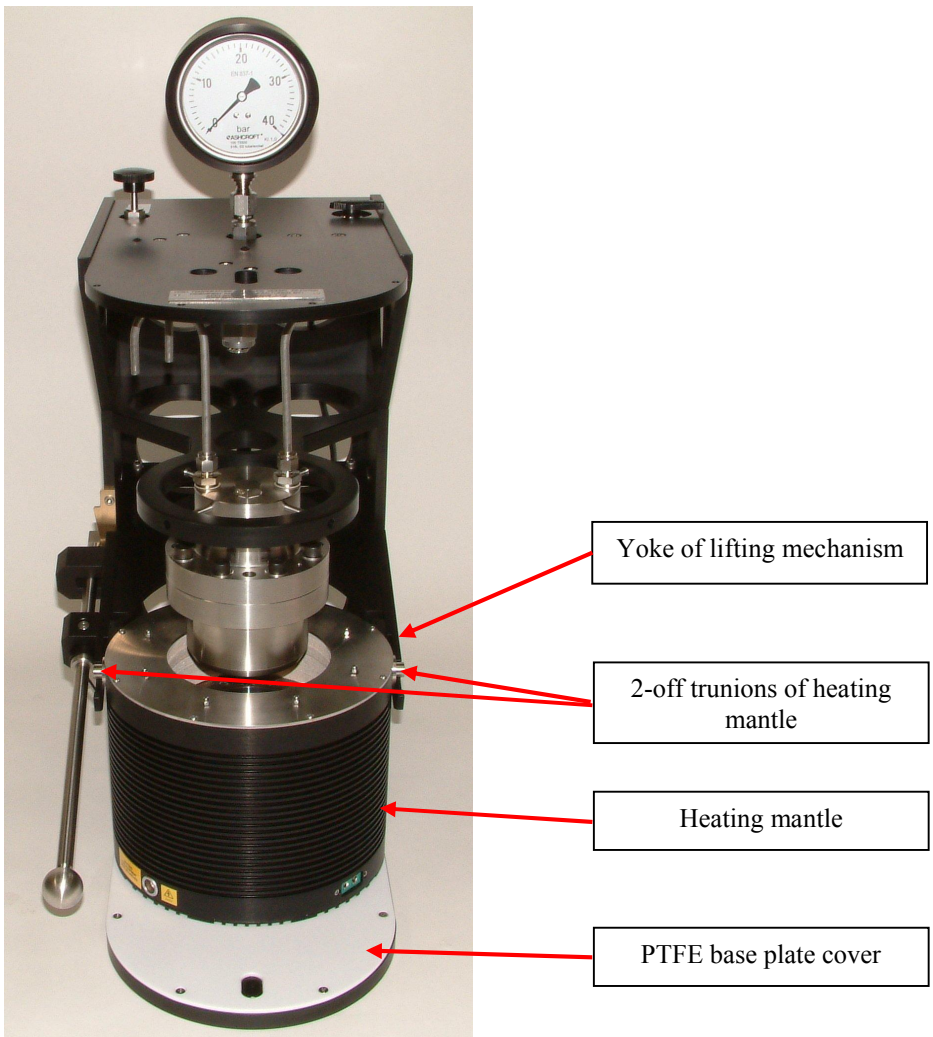

Figure 11. Image of heating mantle correctly aligned with lifting mechanism

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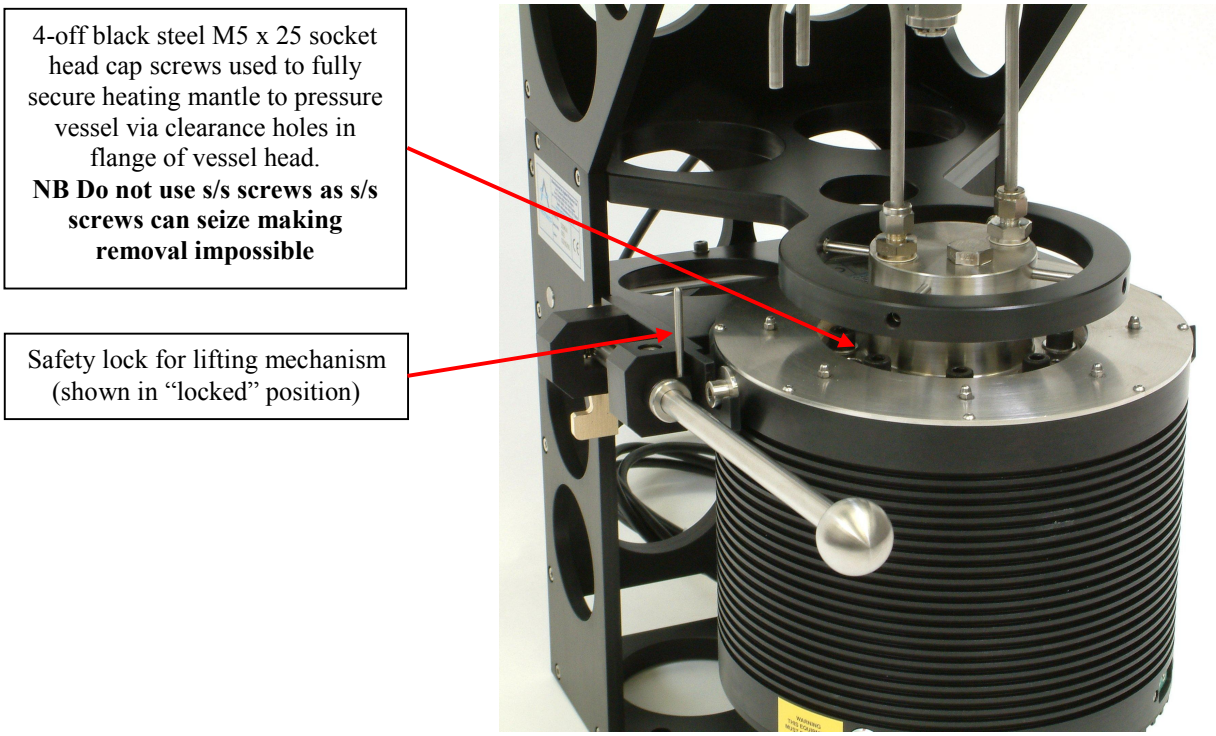
4-off black steel M5 x 25 socket head cap screws used to fully secure heating mantle to pressure vessel via clearance holes in flange of vessel head.
NB Do not use s/s screws as s/s screws can seize making removal impossible
Safety lock for lifting mechanism (shown in "locked" position)

Figure 12. Image of heating mantle lifted into position

4.3 Configuring and operating the Pressure Vessel Controller (PVC)

The PVC may be configured to operate in either of two modes. Normal mode of operation is to use the heating mantle thermocouple to measure the temperature of the pressure vessel (ie. assume that the temperature of the heater body is close to that of the pressure vessel). In this mode, the heating mantle thermocouple is connected to the PID temperature controller (digital display, front panel) using the supplied "plug-to-socket" thermocouple lead. The Heater Protection Controller (analogue display, rear panel) and its associated thermocouple input (front panel) are taken out of circuit by selecting the "0" position on the mode selection switch, as illustrated in figure 13.

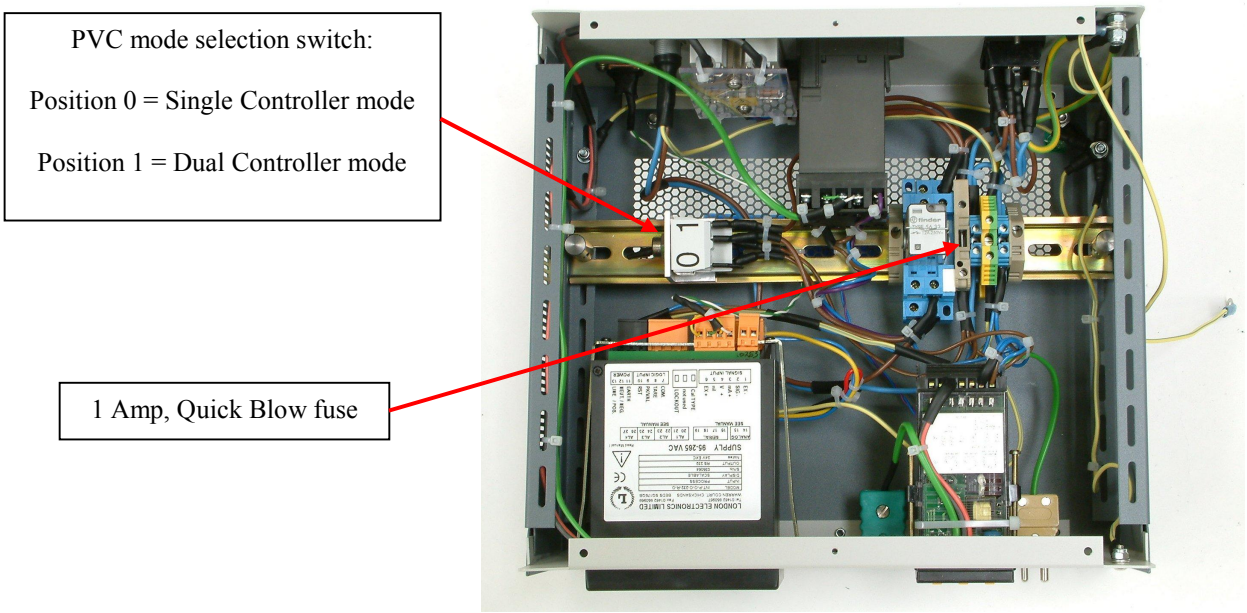

Figure 13. Inside view of PVC showing mode selection switch

If the user wishes to use an alternative thermocouple probe to measure the temperature of the pressure vessel directly, then it will be necessary to configure the PVC to operate in "Dual Controller" mode by selecting Position 1 on the mode selection switch. The alternative thermocouple probe should then be

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connected to the "PID Controller T/C Input" (front panel). In this mode, it is essential that the heaters are protected from overheating and so the heating mantle thermocouple should be connected to the "Heater Controller T/C Input" (front panel) using the supplied "plug-to-plug" thermocouple lead and the "Heater Protection Controller" (rear panel) is set to a temperature of typically 50°C higher than the required temperature of pressure vessel (ie. as set by the "Vessel Temperature" PID controller on the front panel).

Warning:
The "Heater Protection Controller" should never be set to a temperature of greater than 650°C and the "Vessel Temperature" PID controller should never be set to a temperature of greater than 600°C.

Operation of the "Vessel Temperature" PID controller, the "Vessel Pressure" readout controller, and the "Heater Protection Controller" is described in the relevant manufacturer's instructions which are included with the Technical File documentation provided with the High-Temperature Pressure Vessel Reactors.

4.4 Connecting the Pressure Vessel Controller to the heating mantle and pressure transducer

The normal mode of operation for the Pressure Vessel Controller (PVC) is the "Single Controller" mode and the electrical connections to the PVC are as illustrated in figure 14.

Warning
It is strongly recommended that the PVC is connected to the heating mantle before mains power is supplied to the unit.

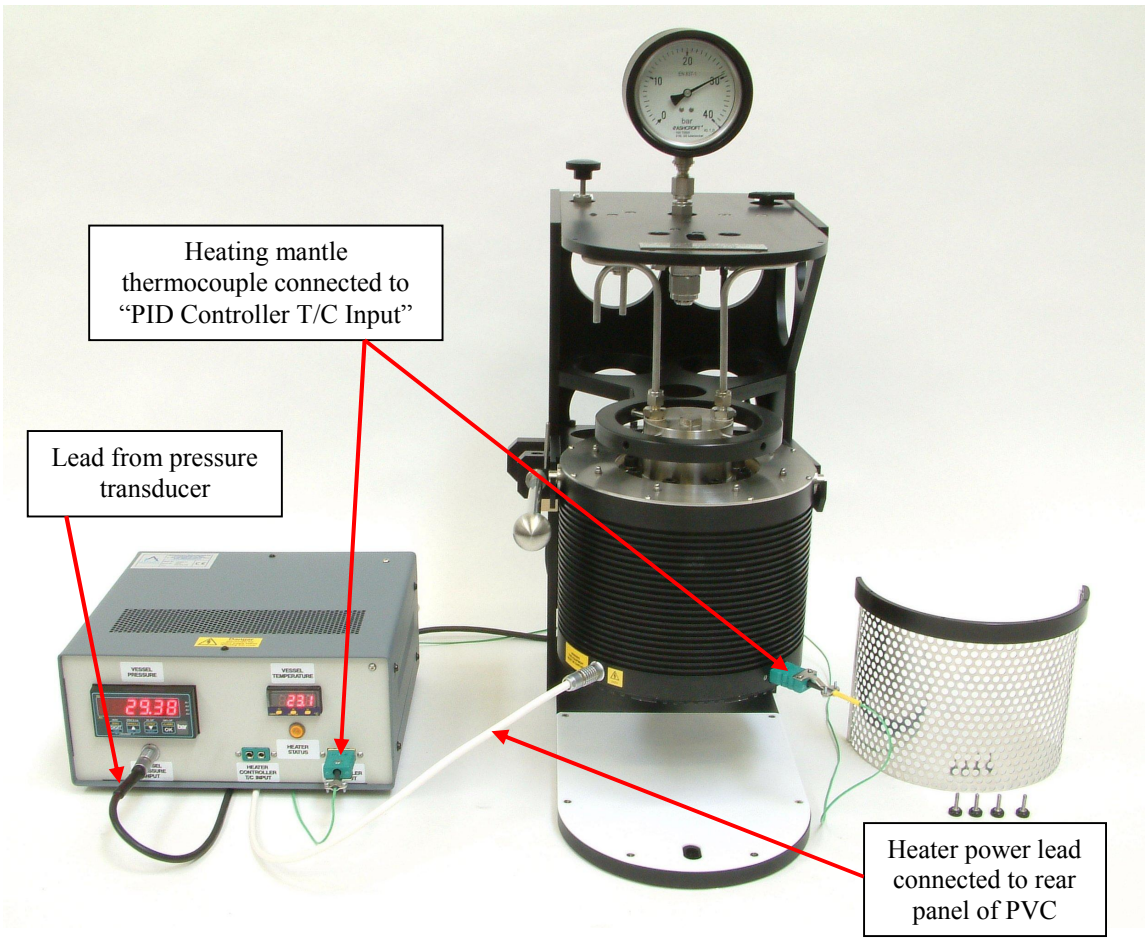
Heating mantle thermocouple connected to "PID Controller T/C Input"
Lead from pressure transducer
Heater power lead connected to rear panel of PVC

Figure 14. Image illustrating the electrical connections between the PVC, heating mantle and pressure transducer when configured in "Single Controller" mode (see section 4.4).

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4.5 Adjusting the pressure relief valve

The units are fitted with two independent safety devices to prevent over-pressurisation of the pressure vessel. The maximum pressure is limited to 37 Bar by the precision burst disc. If this pressure is exceeded, the burst disc will open and allow the gas within the pressure vessel to vent to the outside via a sintered metal filter. It will then be necessary to replace the burst disc.

Warning

It is vitally important that the same type and rating of burst disc is used - contact the manufacturer if a replacement burst disc is required.

The location of the adjustable pressure relief valve is given in figure 15. The cracking pressure of the relief valve is adjustable over the approximate range 20 bar to 50 Bar. It is recommended that the relief valve is set to ~ 30 Bar although to prevent seepage of gas which could interfere with some experiments conducted at near to this pressure, it may be necessary to increase the cracking pressure up to the maximum of ~50 Bar. Under these circumstances, the primary safety relief device will be the burst disc. To reduce the risk of weakening the burst disc which could result in operation of the burst disc at pressures below its rated pressure of 37 Bar, it is important that the user ensures that the vessel pressure does not exceed 30 Bar.

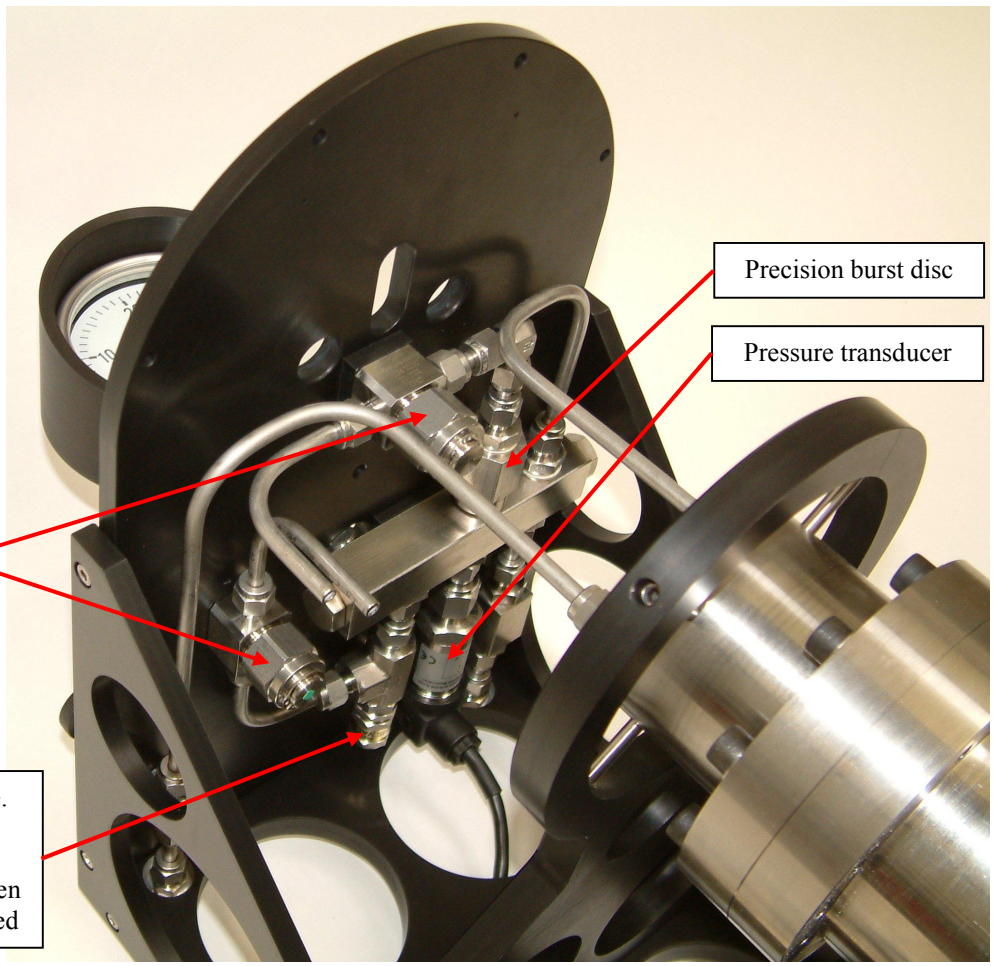
Precision burst disc
Pressure transducer
Sintered metal filters
Adjustable pressure relief valve. To increase cracking pressure, tighten head screw clock-wise Cracking pressure = ~50 Bar when relief valve head is fully tightened

Figure 15. View of pipework showing location of pressure relief valve, burst disc and associated components

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5 Maintenance and inspection

5.1 Pipework and fittings

All pressurised components should be periodically inspected for damage and/or leaks. If a leak is suspected from any of the components, contact the manufacturer for advice on how to repair the leak. If any component appears to be damaged or is malfunctioning in some way, the entire unit should be returned to the manufacturer for servicing.

5.2 Pressure vessel body

The M8 threaded holes (8-off) should be periodically checked for wear using the supplied thread gauge. If the "no-go" end of the thread gauge can be screwed into any of the M8 threaded holes by more than approx. one turn, the pressure vessel should be immediately removed from service and a replacement obtained from the manufacturer.

5.3 Pressure vessel head

Periodically inspect the 4-off attachment pins to the pressure vessel head (ie. modified s/s M8 x 40 grub screws, Pt. No. 1.22) to ensure that they are adequately tight (use an appropriate Allen key to tighten). If any of the attachment pins become bent, contact the manufacturer for a replacement and instructions on how to fit the new part.

5.4 Heating mantle

The heater body (and associated support ring) should be checked before each use to ensure that it is not loose inside the thermal insulation. If it is found to be loose, it will be necessary to tighten the 8-off M3 dome nuts (located on the top cover of the heating mantle) while the heating mantle is fitted to the pressure vessel (to ensure correct alignment of the heater body with respect to the heater mantle). It is important not to over-tighten the M3 dome nuts – they should only be tightened as necessary to adequately secure the heater body. Excessive tightening may permanently damage the internal components of the heating mantle.

The heating mantle should be periodically checked for damage, in particular the thermal insulation discs which are quite fragile. It is acceptable for the insulation discs to have some cracks but they should not be loose or have significant amounts of material missing. If significant damage is observed, the heating mantle should be returned to the manufacturer for servicing.

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Appendix A1

Certificate of Conformity

Applied Photonics Limited
Unit 8 Carleton Business Park
Skipton North Yorkshire
BD23 2DE United Kingdom

EC
Declaration of Conformity

Applied Photonics Ltd declares that the product listed below has been designed and manufactured in compliance with the relevant standards as follows:

Product name: **High-Temperature Pressure Vessel Reactor and ancillary equipment**

Model Number: **0096/A, 0096/B, and 0096/C**

Electrical Safety
This device conforms with the principal safety objectives of the **European Directive 73/23/EEC**, as implemented by the Electrical Equipment (Safety) Regulations 1994, by application of the following standard: **BS EN 61010-1:2001.**

Pressure equipment safety
This device conforms with the principal safety objectives of the **European Directive (97/23/EEC)**, as implemented by the Pressure Equipment Regulations (1999), by application of Sound Engineering Practice (SEP). **NB. CE marking is not required in this instance.**

Electro-Magnetic Compatibility
This device conforms with the principal objectives of the European Directive (89/336/EEC) as amended by 91/31/EEC and 93/68/EEC, as implemented by The EMC Regulations (SI 1992 No. 2372 and amendment SI 1994 No. 3080), by application of the following standard: **BS EN 61326-1:1997**

Year of affixation of the CE Marking: 2005

Signed:

Name: Andrew I. Whitehouse

Title: Managing Director

Place: Applied Photonics Ltd, Unit 8 Carleton Business Park, Skipton, North Yorkshire BD23 2DE, United Kingdom

Date: 31 May 2005

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